

Total Sulfur in Liquid Fertilizers

1.0 Scope

This method determines the total sulfur in liquid fertilizers. Total sulfur in liquid fertilizers includes sulfate, sulfite, thiosulfate and sulfide sulfurs.

2.0 Summary

The sulfur in the sample is oxidized to sulfate with hydrogen peroxide. The resulting sulfate is precipitated as barium sulfate and the weight of the barium sulfate is used to calculate the percent total sulfur in the sample.

3.0 Apparatus and Materials

3.1 500 ml erlenmeyer flask or 400 ml beaker.

3.2 Watch glass.

3.3 Gooch crucible.

3.4 Hot plate.

3.5 Oven, 250°C.

3.6 Whatman No. 42 filter paper.

3.7 Glass fiber filter paper.

4.0 Reagents

4.1 Barium chloride solution (10%): Dissolve 100 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in 900 ml of deionized water. Filter through Whatman No. 42 filter paper.

4.2 Sodium hydroxide Solution (50%): Transfer 50 g NaOH to a 100 ml volumetric flask, dissolve in deionized H_2O , cool and dilute to volume.

- 4.3 Hydrogen Peroxide, 30%. Hydrogen peroxide is hazardous. Read precautions and follow proper procedures for safe handling.
- 4.4 Hydrochloric acid (1 + 1): Combine equal volumes of hydrochloric acid and deionized water. Always add the acid to the water.

5.0 Procedure

- 5.1 Weigh a portion of sample (to the nearest 0.0001 g) containing about 0.5 g of the liquid sample into a 500 ml erlenmeyer flask. Record the weight of the sample.
- 5.2 Add 50 ml of deionized water, 2 ml of 50% sodium hydroxide and 2 ml of 30% hydrogen peroxide to the flask.
- 5.3 Cover the flask with a watch glass and heat gently (refluxing) for 1 hour. Add 1 ml increments of hydrogen peroxide as the reaction subsides. Do not exceed 5 ml of total hydrogen peroxide.
- 5.4 After one hour remove the watch glass and rinse it off into the flask with deionized water.
- 5.5 Bring the volume to about 175 ml with deionized water and add about 10 ml of the (1 + 1) hydrochloric acid.
- 5.6 Bring the solution to a boil. Add slowly, with constant stirring, a slight excess of barium chloride solution (about 15 ml) to the flask.
- 5.7 Digest the sample in the flask on a low temperature hot plate or steam bath for one hour. **Important:** the temperature must be adjusted so that the solution does not boil.
- 5.8 Let stand at room temperature overnight.
- 5.9 Dry a gooch crucible and glass fiber filter paper for a minimum of 60 minutes at 250°C. Cool in a desiccator and weigh to the nearest 0.0001 g.
- 5.10 Filter the cooled solution and BaCl₂ precipitate through the gooch and filter paper.
- 5.11 Wash the precipitate, gooch and filter paper with 10 portions of hot deionized water.

- 5.12 Dry the gooch, filter paper and precipitate at 250°C for a minimum of 60 minutes. Cool in a desiccator and weigh to the nearest 0.0001 g.

6.0 Calculations

- 6.1 Calculate the weight (in grams) of the BaSO₄ precipitate by subtracting the weight of the gooch and filter paper (step 5.9) from the weight of the gooch, filter paper, and precipitate (step 5.12).
- 6.2 Calculate the percent total sulfur using the following equation:

$$\% \text{ Total Sulfur} = \frac{\text{g BaSO}_4 \times 0.1374 \times 100}{(\text{g sample})}$$

7.0 Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., sec. 2.183(b)